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(71) Applicant (for all designated States except US): THE BROKEN HILL PROPRIETARY COMPANY LIMITED [AU/AU]; 600 Bourke Street, Melbourne, VIC 3000 (AU).

(72) Inventor; and
(75) Inventor/Applicant (for US only) : BEEBY, Julie, P. [AU/AU]; 3 Wrexham Circlet, Buttaba, NSW 2283 (AU).

(74) Agent: MUNT, Gregory, Richard; Griffith Hack & Co., 601 St. Kilda Road, Melbourne, VIC 3004 (AU).

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(54) Title: THE RECOVERY OF A VALUABLE SPECIES FROM AN ORE

(57) Abstract

A process for recovery of a valuable species in an ore that contains constituents of absorbed microwave energy is disclosed. In particular, a process for recovery of gold values from a gold bearing ore is disclosed. The process comprises: (a) crushing the gold bearing ore so that up to 95 % of the ore passes a 2 mm screen; (b) exposing the crushed ore to pulses of microwave energy of 1 to 30 seconds duration with intervals of 10 seconds to 2 minutes between the pulses for a total treatment period of up to 1 hour to condition the crushed ore; (c) wet grinding the conditioned crushed ore to a size of -76 μm; and (d) leaching the gold values from the crushed, conditioned, ground ore.

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THE RECOVERY OF A VALUABLE SPECIES FROM AN ORE

The present invention relates to the recovery of a valuable species from an ore that contains constituents that absorb microwave energy.

In particular, the present invention relates to the recovery of gold from gold bearing ores.

In gold bearing ores there are gold metal and compound particles embedded in host rock that may consist of a range of materials such as silica based material, clay or limestone. The gold in these ores is often associated with sulphides, most commonly pyrite and arsenopyrite. Gold recovery from such gold bearing sulphide ores is difficult because there is a high degree of dissemination of the gold content which makes it impossible to achieve an adequate liberation of the gold values by grinding alone. As a consequence, a relatively complicated process is required to recover the gold values. The process comprises treating the gold bearing sulphide ores by flotation techniques to produce a sulphide concentrate, roasting the sulphide concentrate, and finally leaching the gold values from the sulphide concentrate by cyanidation.

An object of the present invention is to provide an improved process for gold recovery from gold bearing ores.

According to the present invention there is provided a process for recovery of a valuable species from an ore

that contains constituents that absorb microwave energy, comprising the steps of:

(a) exposing the ore to pulses of microwave energy of 1 to 30 seconds duration with intervals of 10 seconds to 2 minutes between the pulses for a total treatment period of up to 1 hour to condition the ore; and

(b) recovering the valuable species from the conditioned ore.

Preferably, the pulses are of 2 to 20 seconds, more preferably 2 to 15 seconds, typically 2 to 10 seconds, duration.

Preferably, the intervals between the pulses are 10 to 90 seconds, more preferably 30 to 60 seconds.

Preferably, the total treatment time is up to 40 minutes, more preferably between 5 and 40 minutes, typically between 10 and 40 minutes. In a particularly preferred form of the process the total treatment time is between 20 and 40 minutes.

Preferably, the process comprises a step of crushing the ore prior to exposing the ore to pulses of microwave energy. In a particularly preferred form of the process the ore is crushed so that up to 95% of the ore passes a 2 mm screen.

Preferably, the valuable species is gold and the step of recovering the gold from the conditioned gold bearing ore comprises leaching the conditioned ore by

cyanidation.

Preferably, the process comprises a step of wet grinding the conditioned gold bearing ore prior to leaching the gold values from the conditioned ore.

Preferably, the valuable species are lead and zinc.

The present invention is described further by way of example with reference to the accompanying drawings in which:

Figure 1 is a diagram of an apparatus used to carry out experimental work to evaluate the process of the present invention;

Figure 2 is a plot of experimental results which illustrate the relationship between gold recovery and microwave energy exposure time for ore samples treated in accordance with the process of the present invention; and

Figure 3 is a further plot of experimental results which illustrate the relationship between gold recovery and microwave energy exposure time for ore samples treated in accordance with the process of the present invention.

The present invention is based on the finding that recovery of valuable species, such as gold, lead and zinc, that are difficult to separate from an ore can be improved by exposing the ore to pulsed microwave energy to cause mechanical breakdown of the ore.

In particular, the present invention is based on the finding that gold values can be recovered from a gold

bearing ore in a process which does not require flotation and roasting steps but which includes the steps of, exposing the ore, preferably after crushing the ore, to pulses of microwave energy of 1 to 30 seconds duration with intervals of 10 seconds to 2 minutes between pulses for a total treatment time of up to 1 hour to condition the ore, and subsequently leaching the gold values from the conditioned ore, preferably after wet grinding the conditioned ore to a size of -76 μm.

The present invention is described hereinafter with references to the following Examples.

EXAMPLE 1

A series of experiments was carried out on a first sample of lump "rock" gold bearing ore with exposed sulphide mineralization obtained from Ora Banda gold mine to illustrate the process of the present invention.

The gold content of the ore was determined to be relatively low, 1.40 ± 0.38 mg kg^{-1}, and it was found from X-ray diffraction analysis that the ore had the constituents set out in Table 1 below.

Constituent	Proportion in ore
Quartz	Low (3-10%)
Illite	Present (10-40%)
Chlorite	Present (10-40%)
Feldspars	Very low (0.3-3%)

Calcite	Low (3-10%)
Dolomite	Very low (0.3-3%)
Pyrite	Very low (0.3-3%)
Pyrrhotite	Very low (0.3-3%)
Arsenopyrite	Very low (0.3-3%)

The ore was assessed for gold leachability by leaching 20 g samples of ore, crushed and subsequently wet ground to -76μm, in alkaline cyanide solution (0.005 M NaCN (800 ml) pH 10.5 leaching solution, agitated at 22° C) for up to 24 hours. Leachate samples were removed at regular intervals for gold content analysis. The quantity of gold leached was 36.6 ± 4.9% of the gold present in the ore. This figure suggests a high (13%) variation in the leachability of the ore which is believed to be a result of heterogenity of the gold distribution in the ore.

A 12 kg sample of the ore was reduced to a size of -10 mm. This size approximates the size to which Ora Banda ore is crushed before it is introduced to a ball mill for wet grinding. A size analysis of the crushed ore is shown in Table 2 below.

Size fraction (mm)	% in size range	Cum. % passing topsize
-16+8	11.81	100.00
-8+4	36.01	88.19
-4+2	15.83	52.18
-2+1	10.67	36.35
-1+0.5	6.35	25.68
-0.5+0.25	3.20	19.33
-0.25+0.125	1.87	16.13

-0.125+0.063	2.26	14.26
-0.063	12.00	12.00

The crushed ore was treated in accordance with the process of the present invention, in a batch mode, in the experimental apparatus shown in figure 1. It is noted that the process of the present invention is not restricted to treatment of ore in a batch mode and extends to treatment in a continuous mode.

With reference to Figure 1, the experimental apparatus comprises a microwave oven 1 and a glass reactor 3 designed to hold a 200 g sample of ore and arranged to rotate in the microwave oven 1 in the direction indicated by the arrow at a rate of 10 rpm. The apparatus further comprises an inlet 4 for injection of air or nitrogen into the glass reactor 3 and an outlet 5 for discharging gases from the glass reactor 3. The apparatus further comprises a filter (glass fiber wool) to remove any solid materials entrained in the gases positioned in the outlet 5 and a sodium hydroxide trap 7 to absorb any evolved acidic gases located downstream of the outlet 5. The apparatus further comprises a thermocouple 9 to monitor the temperature of the ore.

The microwave oven 1 used was a Sharp R-2370 with two 650 W magnetrons that could be used singly or together. The microwave energy was generated at a frequency of 2.45 GHz. The microwave energy did not have a variable control of the magnetrons and thus could only deliver microwave energy at 650 W or 1300 W. The

magnetrons were set to be active for a certain percentage of time every 30 seconds, ie, on a 20% setting the magnetron operated for 6 consecutive seconds every 30 seconds. In other words, the magnetrons were set to deliver pulsed microwave energy.

A series of samples of the crushed ore was exposed to pulsed microwave energy in the apparatus shown in Figure 1 over a range of microwave energy power and exposure settings. The microwave energy processed samples were assessed for gold leachability by wet grinding 20 g of each sample to -76 μm and leaching each 20 g ground sample in 800 ml of alkaline sodium cyanide solution for 24 hours in accordance with the procedure described above for assessing the leachability of the unprocessed ore samples.

The results of the gold leachability for unprocessed samples and for a series of microwave energy processed samples exposed for up to 40 minutes at a microwave energy power level of 650 W at settings of 10%, 20% and 40% are shown in Figure 2. It can be seen from the figure that after 10 minutes of processing the leachability of the gold decreased from that of unprocessed ore, but 25 minutes processing brought about an increased leachability in all cases. The reason for this is not known and the results, although consistent, are considered to be surprising. It is also evident from the decrease in leachability that occurred between 25 and 40 minutes processing time for each of the samples shown in the figure that the ore can be over-processed.

The results of the gold leachability for unprocessed samples and for a series of microwave energy processed

samples exposed for up to 40 minutes at microwave energy power levels of 650 W and 1300 W at 20% and 10% settings, respectively, which produced the same total microwave energy exposure, are shown in figure 3. It can be seen from Figure 3 that the gold leachability achieved with the 10% 1300 W microwave energy samples was better than that achieved with the 20% 650 W microwave energy samples. In this regard, the maximum gold extraction for the 10% 1300 W microwave energy samples, which was 62% higher than that for unprocessed samples, was obtained after only 10 minutes exposure to microwave energy, after which the leachability of the ore decreased dramatically, whereas the maximum gold extraction for the 20% 650W microwave energy samples, which was 42% higher than that for unprocessed samples, required 25 minutes exposure to microwave energy.

It was found from experimental work on the samples with the highest microwave energy processing (40% 650 W, 40 minutes) that there was a strong reaction, resulting in a microwave energy induced plasma in the 26th minute in one section of the ore. The plasma was only sustained while the magnetrons were operating and no reaction was observed in other parts of the ore. The plasma was considered to be due to the presence of a sulphur-based gas. The ore was examined after processing and all particles were found to be fused together. The section was examined by both reflected light microscopy and scanning electron microscopy. The fused area was found to be centred around an atypical concentration of relatively coarse pyrrhotite with minor associated chalocopyrite and ilmenite. The pyrrhotite had developed fine porosity and small prills of metallic iron (formed by localized reduction of the sulphide) were abundant in

some crystals. The K-feldspar, quartz, etc, in the rock matrix adjacent to the pyrite had been transformed into dark Fe-Ti-rich K-Al-silicate glass with variable but minor amounts of calcium and magnesium. The formation of the K-Al-silicate indicated that the localized temperature of the area reached greater than 900°C but the presence of unaffected silica in the glass indicated the temperature did not rise above 1700°C. From this it can be seen that the temperature retained by the pyrite was greater than that required for the process of the present invention. Moreover, the direct reduction of the pyrite to metallic iron is considered to be undesirable because the iron would be leached out with the gold during cyanidation. These results indicate that the microwave energy used in this instance was in excess of that required and this conclusion was substantiated by lowered gold extraction.

The results of an X-ray diffraction analysis of the microwave energy processed samples indicated that there was no significant difference between the crushed ore samples before and after microwave energy processing in accordance with the present invention.

Specifically, it was found from the X-ray diffraction analysis that the quartz, illite, chlorite, feldspars and calcite (non-conductive constituents) in the ore remained unaffected by microwave processing and that the dolomite, pyrite, pyrrhotite and arsenopyrite (conductive constituents) in the ore were all affected to a minor degree by the microwave energy processing and in some instances haematite and magnetite were formed.

The results of a total gold and sulphur analysis of

all of the unprocessed samples and microwave energy processed samples invention indicated that only limited oxidation of the sulphide minerals occurred. The gold content in individual samples had a standard deviation of 27% and a total sulphur content also varied greatly between the samples. However, a plot of the gold versus sulphur content showed a positive correlation between the total gold and sulphur in each sample. If it can be assumed that the gold to sulphur ratio throughout the ore was reasonably constant, it follows from the positive correlation that the microwave energy treatment did not release the sulphur from the ore as would be expected if oxidation had occurred. As a consequence, it is postulated that the improvement in the gold recovery in the samples processed in accordance with the present invention was due to a large extent to mechanical mechanisms resulting from exposure to microwave energy. In this regard, it is thought that pulsed microwave energy results in differential thermal expansion in the crushed ore as a result of differential microwave energy absorption by constituents in the ore, the creation of high temperature internal gas phases in the crushed ore, and crystallographic transformation of constituents in the ore, and all of the foregoing factors induce stress and cracks that result ultimately in structural distortion and breakdown of the crushed ore.

In summary, the experimental results indicated that exposure of Ora Banda ore to pulsed microwave energy in accordance with the present invention could result in an increase of up to 62% of the amount of gold recovered from the crushed ore compared with unprocessed ore. This result was achieved when the crushed ore was treated with 1300 W microwave energy for 3 seconds every 30

seconds for 10 minutes. The examination of the crushed ore before and after microwave energy processing revealed little change in the composition and in particular there was a relatively small amount of oxidation. As a consequence, it is thought that the improvement in gold recovery of the crushed ore was not due to oxidation of sulphides to any significant extent and was due principally to mechanical mechanisms in the crushed ore due to exposure to pulsed microwave energy.

It is noted that the low sulphide oxidation has important implications for reduced sulphur emissions in comparison to conventional roasting techniques. There would also seem to be some potential for reduced arsenic emission as a result of minimal oxidation of arsenopyrite during microwave energy processing in accordance with the present invention.

EXAMPLE 2

A series of experiments were carried out on a second sample of refractory gold ore from the Ora Banda gold mine. The second sample was from a different section of the ore body to that of Example 1.

The ore was assessed for gold leachability by leaching 500 g samples of ore crushed and subsequently wet ground to -76 μm in alkaline cyanide solution (0.02-0.01 M NaCn (500 ml) pH 10.5 leaching solution agitated by 22°C) for up to 24 hours. Leachate samples were removed at regular intervals for gold content analysis. The quantity of gold leached was 45% of the gold present in the ore.

A 100 g sample of the ore was crushed to a size of -10 mm and exposed to 500 W microwave energy for 1 minute in experimental apparatus shown of the type in Figure 1 to study the effect of the timing of microwave exposure on gold recovery. In the context of the power level and the sample size, the period of 1 minute was considered to be "continuous" exposure and significantly longer than what would be the duration of a pulse in accordance with the present invention. It was found that the gold recovery of the sample, after wet grinding to -76 μm and leaching in accordance with the procedure in the preceding paragraph, was reduced to 17% and this indicates that continuous exposure is detrimental to gold recovery.

A number of samples of the ore were crushed to a size of -10 mm and exposed to 500 W pulsed microwave energy to investigate the limits of effectiveness of pulsed microwave energy. A series of experiments was carried out with pulses of 2 to 10 seconds duration and intervals of between 30 and 60 seconds between pulses and total treatment times up to 1 hour. The conditioned samples were wet ground to -76 μm and leached in accordance with the procedure described previously to determine the gold recovery. Generally, good gold recoveries were obtained with all combinations of these times tested. In particular, it was found that the best gold recovery was obtained after total treatment times in the range of 20 to 40 minutes. For example, when 300 g of crushed ore was treated with 500 W microwave energy for a sequence consisting of 6 seconds of irradiation followed by 30 seconds of no radiation repeated over a treatment time of 27 minutes the gold recovery was 63%.

A series of experiments was carried out to investigate whether the effectiveness of pulsed microwave energy in accordance with the present invention is dependent on the frequency of the microwave energy. The experiments were carried out at 915 MHz using a 30 kW magnetron and the results were compared with the results discussed in Example 1 which were obtained using magnetrons operating at 2.45 GHz. The experiments were carried out on 1.5 kg samples of ore crushed to a size of - 10 mm at power settings between 7.5 kW and 22.5 kW. The experiments were carried out with pulses of 2 to 10 seconds duration and intervals of between 30 and 60 seconds between pulses and total treatment times up to 1 hour. The conditioned samples were wet ground to -76 μm and leached in accordance with the procedure described previously to determine the gold recovery. It was found that the gold recovery was dependent on the power levels and the pulse duration and time intervals between pulses. Specifically, it was found that the best results were obtained with total treatment times between 20 and 40 minutes. For example, a sample irradiated with pulses of 7.5 kW microwave energy of 10 seconds and 60 seconds intervals between pulses for a total treatment time of 31.5 minutes resulted in a gold recovery of 55%. With good results obtained at both the 2.45 GHz and 915 MHz frequencies it is believed that the process of the present invention is not dependent on frequency of the microwave energy.

In summary, the experimental results further supported the importance of the use of pulsed rather than continuous microwave energy in the process of the present invention and established that good results could be obtained at microwave energy frequencies of 915 MHz and 2.45 GHz.

EXAMPLE 3

A series of experiments was carried out on oxidized samples of a complex lead-zinc ore. From the results of the experiments it is clear that pulsed microwave energy in accordance with the process of the present invention acts on the ore by an essentially mechanical mechanism of inducing microcracking in and around the microwave absorbing constituents in the ore. The experiments comprised crushing the ore to a size of -8 mm, exposing 200 g samples of the crushed ore to pulses of 650 W or 1300 W microwave energy of 3 or 6 seconds with 30 seconds intervals between pulses, wet-grinding the crushed, conditioned ore to 80% passing 53 m, and separating lead-and zinc-bearing components from the crushed, conditioned ground ore.

Many modifications may be made to the process of the present invention as described in the Examples without departing from the spirit and scope of the present invention.

CLAIMS:

1. A process for recovery of a valuable species from an ore that contains constituents that absorb microwave energy, comprising the steps of:

(a) exposing the ore to pulses of microwave energy of 1 to 30 seconds duration with intervals of 10 seconds to 2 minutes between the pulses for a total treatment period of up to 1 hour to condition the ore; and

(b) recovering the valuable species from the conditioned ore.

2. The process defined in claim 1, wherein the pulses are 2 to 20 seconds duration.

3. The process defined in claim 2, wherein the pulses are 2 to 15 seconds duration.

4. The process defined in claim 3, wherein the pulses are 2 to 10 seconds duration.

5. The process defined in any one of the preceding claims, wherein the intervals between the pulses are 10 to 90 seconds.

6. The process defined in claim 5, wherein the intervals between the pulses are 30 to 60 seconds.

7. The process defined in any one of the preceding claims, wherein the total treatment time is up to 40 minutes.

8. The process defined in claim 7, wherein the treatment time is between 5 and 40 minutes.

9. The process defined in claim 8, wherein the treatment time is between 10 and 40 minutes.

10. The process defined in claim 9, wherein the treatment time is between 20 and 40 minutes.

11. The process defined in any one of the preceding claims, comprising a step of crushing the ore prior to exposing the ore to pulses of microwave energy.

12. The process defined in claim 11, comprising crushing the ore so that up to 95% of the ore passes a 2 mm screen.

13. The process defined in any one of the preceding claims, wherein the valuable species is gold.

14. The process defined in claim 13, wherein the step of recovering the gold from the conditioned ore comprises leaching the conditioned ore by cyanidation.

15. The process defined in claim 14, comprising a step of wet grinding the conditioned ore prior to leaching the gold values from the conditioned ore.

16. The process defined in claim 13, comprising wet grinding the conditioned ore to a size of -76 µm.

17. The process defined in any one of claims 1 to 13, wherein the valuable species are lead and zinc.

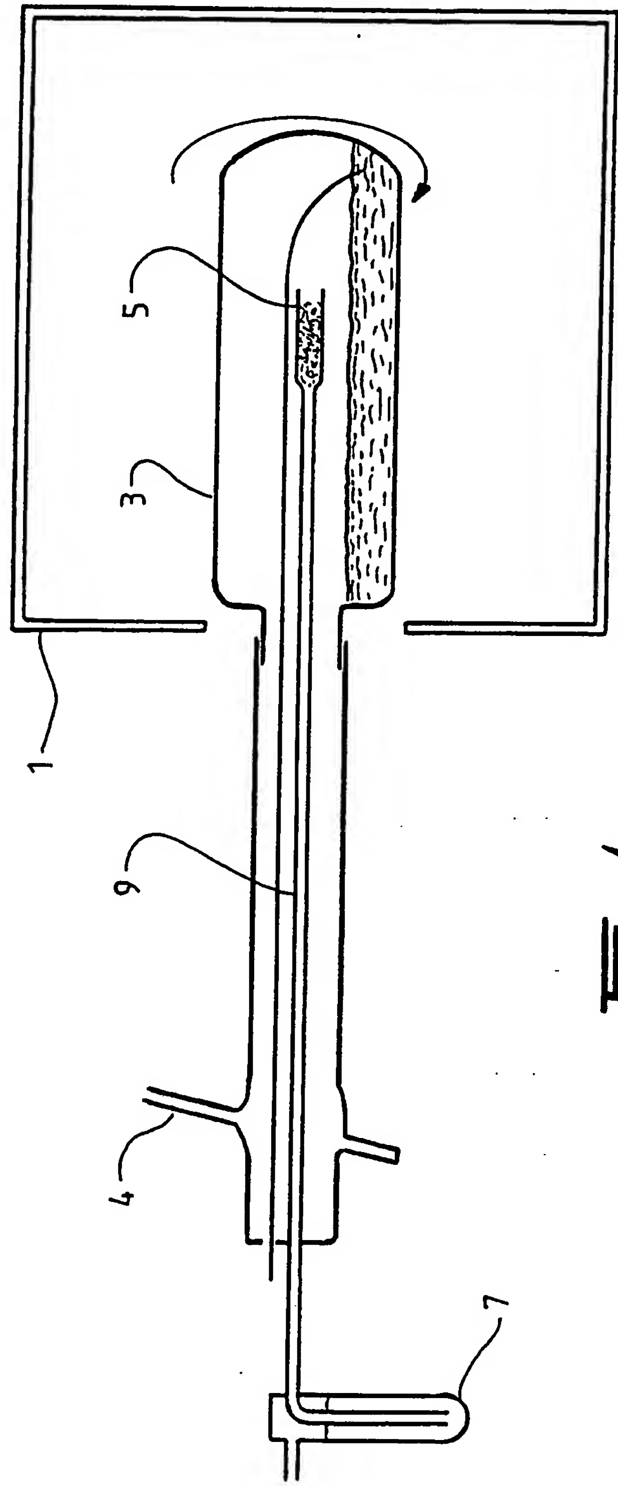
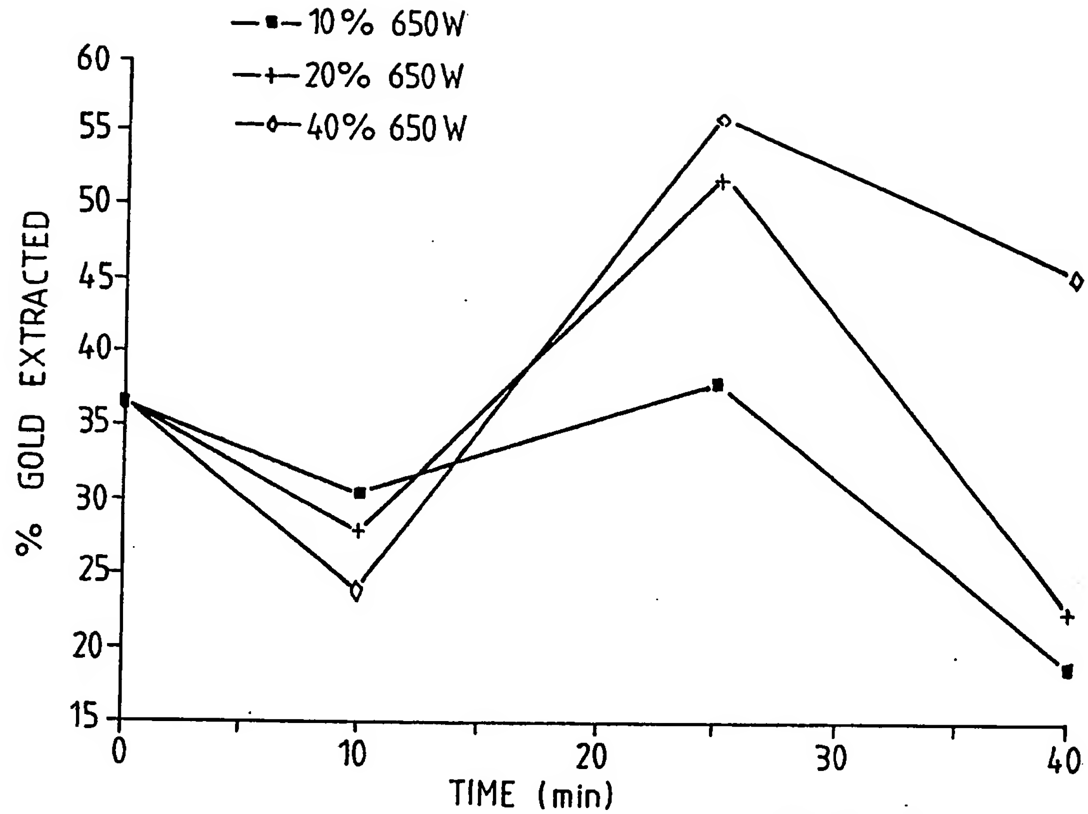
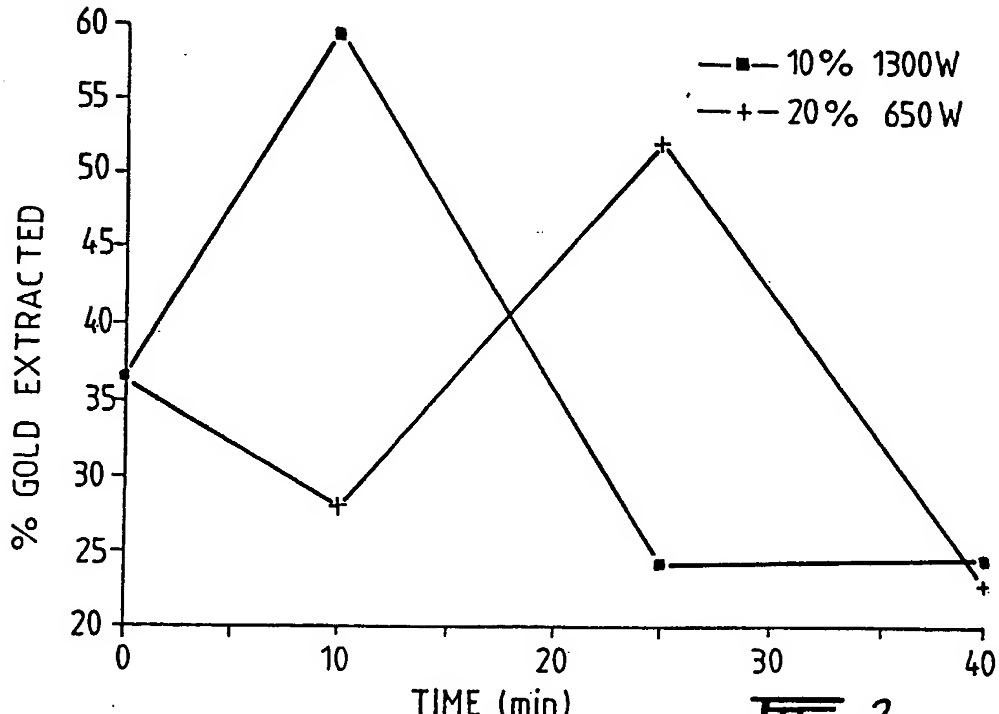


FIG. 1.



—■— 10% 650W
—+— 20% 650W
—◇— 40% 650W
% GOLD EXTRACTED
60
55
50
45
40
35
30
25
20
15
0
10
20
30
40
TIME (min)

FIG. 2.



—■— 10% 1300W
—+— 20% 650W
% GOLD EXTRACTED
60
55
50
45
40
35
30
25
20
0
10
20
30
40
TIME (min)

FIG. 3.

INTERNATIONAL SEARCH REPORT

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)[6]

According to International Patent classification (IPC) or to both National Classification and IPC

Int. Cl.[5] B02C 19/18, C22B 1/00 // C22B 11/00, 3/04

II. FIELDS SEARCHED

Minimum Documentation Searched [7]

Classification System	Classification Symbols
IPC	B02C 19/18, C22B 1/00

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched [8]

AU : IPC as above

III. DOCUMENTS CONSIDERED TO BE RELEVANT [9]

Category*	Citation of Document, [11] with indication, where appropriate of the relevant passages [12]	Relevant to Claim No [13]
Y	AU,B, 8795/61 (256354) (HADEN) 2 May 1963 (02.05.63) Pages 9-10	(13-14)
A	AU,A, 49054/79 (PENNELL) 7 February 1980 (07.02.80)	
A	Derwent Abstract Accession no. 88-069337/10, Class P41, SU,A, 1326334 (AS UKR GOETECH MECH INST) 30 July 1987 (30.07.87)	
X Y	US,A, 4906290 (WORNER) 6 March 1990 (06.03.90) Examples 1-3	(1-3,5-12) (13-14)
A	WO,A, 89/04379 (WORNER) 18 May 1989 (18.05.89)	
X Y	US,A, 4321089 (KRUESI et al) 23 March 1982 (23.03.82) Example 5	(1,12) (13-14)
A	US,A, 4311520 (KRUESI et al) 19 January 1982 (19.01.82)	

* Special categories of cited documents : [10]

"A" Document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior t the international filing date but later than the priority date claimed

"T" Later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
17 June 1992 (17.06.92)	24 June 1992 (24.06.92)
International Searching Authority	**Signature of Authorized Officer**
AUSTRALIAN PATENT OFFICE	B. BOURKE B Burke

FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET

V. ☐ OBSERVATIONS WHERE CERTAIN CLAIMS WERE FOUND UNSEARCHABLE [1]

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claim numbers ..., because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claim numbers ..., because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☐ Claim numbers ..., because they are dependent claims and are not drafted in accordance with the second and third sentences of PCT Rule 6.4a

VI. ☐ OBSERVATIONS WHERE UNITY OF INVENTION IS LACKING [2]

This International Searching Authority found multiple inventions in this international application as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims of the international application.
2. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims of the international application for which fees were paid, specifically claims:

3. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claim numbers:

4. ☐ As all searchable claims could be searched without effort justifying an additional fee, the International Searching Authority did not invite payment of any additional fee.

Remark on Protest

☐ The additional search fees were accompanied by applicant's protest.

☐ No protest accompanied the payment of additional search fees.

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL APPLICATION NO. PCT/AU 92/00162

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report	Patent Family Member		
US 4906290	AU 15299/88 JP 1052028	DE 3814451 SE 8801571	GB 2205559
WO 89/04379	AU 25594/88		
US 4321089	CA 1175240	EP 41841	JP 57067138
US 4311520	AU 67914/81 PH 17184	CA 1160057	FR 2477181

END OF ANNEX